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(54) Title: PECTIN FOR USE IN PASTE–LIKE MATERIALS, A METHOD OF PREPARING THE SAME, PASTE–LIKE MATERIALS COMPRISING THE PECTIN AS WELL AS THE USE THEREOF

(57) Abstract

Pectin for use in paste–like materials has a molecular weight in the range of 20,000 to 50,000 Daltons and a degree of esterification of less than approximately 20, and can be used alone or in combination with a secondary pectin with a molecular weight in the range of from approximately 50,000 to approximately 150,000 Daltons and a degree of esterification of less than approximately 20. The pectin or pectin combination, being soluble in ordinary tap water, can be used in products which are pasty and heat stable, such as in bake stable jams and milk desserts.

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Title: Pectin for use in paste-like materials, a method of preparing the same, paste-like materials comprising the pectin as well as the use thereof.

Technical Field

The invention relates to a pectin for use in paste-like materials, paste-like materials comprising the pectin as well as the use thereof.

Background Art

Pectins can be used for the production of a number of foodstuffs, such as jam, jelly and fillings for pies etc., including heat tolerating foodstuffs such as by baking at up to approximately 200 to 250°C for up to 20 minutes without "flowing".

Industrially produced pectins are made up primarily of polygalacturonic acid chains in which rhamnose may be found. In addition, neutral sugars may be attached to the rhamnose units. The anhydrogalacturonic acid makes up at least 65% of the solids in commercial pectins. The galacturonic acids are partly esterified with methyl alcohol.

According to convention, pectins with more than 50% of the carboxylic acid groups esterified with methyl alcohol are referred to as high methoxyl pectins, whereas pectins with less than 50% of the carboxylic acid groups esterified with methyl alcohol are called low methoxyl pectins.

In the present description the expression "degree of esterification" means the degree in which the free carboxylic acid groups in the polygalacturonic acid chain have been esterified by way of methylation. In other words, the degree of esterification is defined as the number of esterified galacturonic acid units expressed in percentages of all the galacturonic acid units in the molecule, and accordingly it assumes a value between 0 and 100%.

The pectins hitherto used for heat stable foodstuffs comprise both pectins with a high degree of esterification and pectins with a low degree of esterification. Pectins with a high degree of esterification (DE) are characterised by resulting in a very strong and hard texture requiring a mechanical processing (a vigorous stirring) before they can be pumped or spread. The mechanical processing has, however, the effect that these pectins with a high DE cause a separation of water (syneresis) and consequently a reduced bake stability. Pectins with a low degree of esterification are characterised by being pumpable and spreadable, but in order to make them heat stable it is necessary to reduce the degree of esterification to below approximately 10 with the result that difficulties arise in connection with the dissolving of the pectin in hard water with degrees of hardness exceeding approximately 10°H (71.5 ppm calcium), and that a partial dissolving in distilled water is necessary.

Brief Description of the Invention

It turned out surprisingly that it is rendered possible by the use of a novel pectin with a molecular weight in the range of approximately 20,000 to 50,000 Daltons and a degree of esterification of less than approximately 20, preferably less than approximately 10, especially less than approximately 5, as the sole gelling agent or in combination with a pectin with a higher molecular weight and a degree of esterification of less than approximately 20, preferably less than approximately 10, and especially less than approximately 5, to achieve products which simultaneously are both pasty and heat stable. In addition the advantages are obtained that it is possible to use ordinary tap water for dissolving the pectin, and that a pasty texture is achieved without involving a vigorous stirring.

In the present description the expression "gelling agent" refers to an agent characterised by resulting in a structure being either solid or viscous.

When a combination of a low-molecular pectin and a high-molecular pectin is used, it is possible to tailor the pectin combination by varying the ratio of the low-molecular weight pectin, viz. the so-called primary pectin, to the higher molecular weight pectin, viz. the so-called secondary pectin, such that said combination has an optimum effect within various fields of application, such as in bake-stable jams with a high solids content and in pH-neutral "fillings" with a high solids content.

Therefore a first object of the invention is a pectin being characterised by having a molecular weight in the range of from approximately 20,000 to approximately 50,000 Daltons and a degree of esterification (DE) of less than approximately 20.

Another object of the invention is a method of producing the pectin according to the invention, said method being characterised by obtaining the pectin in a manner known per se followed by said pectin being subjected to molecular weight reduction as well as a reduction of the degree of esterification.

The pectin according to the invention can be produced from any conventional pectin raw material, such as from grape, lemon, lime or orange peel, from which the pectin is obtained in a conventional manner. A typical method involves the steps:

1) acid extraction from the plant starting material at a low pH value,

2) purification of the liquid extract, and

3) isolation of the extracted pectin from the liquid.

In this connection reference is made to EP publication No. 664,300 A1 disclosing methods of producing pectin.

Thereafter the pectin obtained is subjected to an additional treatment in order to reduce the molecular weight from approximately 60,000 - 160,000 Daltons, preferably from approximately 80,000 - 120,000, to the desired range of approximately 20,000 - 50,000 Daltons, preferably approximately 25,000 - 40,000 Daltons, and in order to reduce the degree of esterification from approximately 55 - 75%, preferably from 55 - 65%, to less than approximately 20%, preferably less than approximately 10%, and especially less than approximately 5%.

The reduction of the degree of esterification can be performed by way of alkaline treatment, such as by means of an alkali metal hydroxide, such as NaOH, KOH or LiOH, or by means of an alkaline earth metal hydroxide, such as $Ca(OH)_2$ or $Mg(OH)_2$. The reduction of the degree of esterification can also be performed enzymatically, optionally by means of pectin methyl esterase. Moreover, the degree of esterification can be reduced by combining an acid treatment by means of an inorganic acid, such as HNO_3, HCl or H_2SO_4, preferably HNO_3, with an alkaline treatment and/or an enzymatic treatment.The degree of esterification is titrimetrically determined, for instance by means of the FCC and FAO/WHO method, cf. Food Chemicals Codex, FCC IV Monographs, 4th. ed., National Academy Press, Washington DC, 283 (1996) as well as FAO, Food and Nutrition Paper 52. Addendum 1, Rome, 87 (1992).

The molecular weight reduction can be performed enzymatically or by means of base. When the molecular weight reduction is performed by means of a base, an alkali metal hydroxide, such as NaOH, KOH or LiOH, preferably NaOH, or an alkaline earth metal hydroxide, such as $Ca(OH)_2$ or $Mg(OH)_2$ is preferably used. The reduction of the molecular weight can also be performed enzymatically by means of for instance endopolygalacturonase or pectin lyase or alternatively pectate lyase. When pectate lyase is used, it is assumed that the degree of esterification has been reduced prior to the pectate lyase treatment.

The molecular weight can be determined by way of glass capillary viscosimetry, whereby the relative viscosity of an 0.1% pectin solution is measured by means of Na-hexametaphosphate, jf. Christensen, P.E.: "Methods of Grading Pectin in Relation to the Molecular Weight (Intrinsic Viscosity) of Pectin", Food Research, Volume 19, pages 163 through 171 (1954).

The molecular weight reduction as well as the reduction of the degree of esterification can be performed in one or more steps. When a two-step process is followed, it is often chosen to reduce the degree of esterification first followed by the molecular weight reduction, but the order is not decisive.

The reduction of the degree of esterification and/or the molecular weight can be performed between steps 2 and 3 in the process for obtaining pectin as described above, cf. EP publication No. 664,300 A1, and/or after step 3.

The pectin according to the invention can be used either alone or in combination with a high-molecular pectin for the production of paste-like materials.

Therefore the invention also relates to a paste-like material comprising as gelling agent a pectin according to the invention.

According to a preferred embodiment of the invention, the paste-like material comprises a combination of least one pectin according to the invention, viz. the primary pectin, and at least one secondary pectin with a molecular weight in the range of approximately 50,000 to 150,000 Daltons and a degree of esterification of less than approximately 20. The secondary pectin has preferably a degree of esterification of less than approximately 10, especially of less than approximately 5. In addition, the

secondary pectin has preferably a molecular weight in the range of 60,000 to 110.000 Daltons, especially approximately 80,000 to 90,000. An example of a secondary pectin is GENU Pectin type LM-5 CS, produced by Copenhagen Pectin A/S.

When a combination of a primary pectin and a secondary pectin is used, the ratio of the primary pectin to the secondary pectin is in the range of from approximately 5:1 to approximately 1:3, preferably from approximately 3:1 to approximately 1:1.

The use of a combination of a primary pectin and a secondary pectin renders it possible by way of a control of the mutual proportional ratio to tailor the paste-like material to a specific use, such as for bake stable jams with a high solids content and produced on the basis of any desired fruit, such as strawberry, raspberry, apple, apricot or the like fruit.

According to another object the invention therefore relates to the use of a paste-like material in a fruit-based product, preferably in an amount in the range of 0.8 to 1.5% by weight, especially approximately 1.0 to 1.2% by weight.

Furthermore, the paste-like material can be used for neutral fillings, such as milk-based creams with for instance vanilla or caramel taste, preferably in an amount in the range of 0.8 to 1.5% by weight, especially approximately 1.0 to 1.2% by weight.

In addition to the above fields of application the invention can also be used for other types of foodstuffs desired to be gelled, such as vegetable pastes, for instance tomato paste, or meat or fish-based products, such as pâté.

The ratio of the primary to the secondary pectin varies generally in

response to the solids content in the final product. Thus, high solids contents (SS = soluble solids) of about 75% involve the use of a relatively high proportion of primary pectin, whereas lower solids contents involve the use of a comparatively lower proportion of primary pectin.

Here it should be noted that as a (partial) replacement or supplement of the secondary pectin it is possible to vary the Ca^{2+}-content in the final product so as to adjust the product characteristics. The latter is carried out by adding an amount of a food quality calcium salt corresponding to a calcium amount of 0 to 500 ppm Ca^{2+}, preferably approximately 50 to 200 ppm Ca^{2+}, taking into account the natural calcium-content of the foodstuff in question.

The invention is illustrated below by means of the following Examples.

EXAMPLES

Example 1

Preparation of primary pectin.

a) Molecular weight reduction

4.035 kg of liquid extract of citrus peels comprising 36.6 g of dry pectin with a molecular weight of approximately 100,000 and a degree of esterification DE of approximately 62 are introduced in a tube reactor and adjusted at a pH value of 4.0 by addition of Na_2CO_3. The input temperature is adjusted at 47°C ± 2°C, whereafter a solution of endopolygalacturonase commercialized under the trade name Rohament P by the company Röhm is added in a concentration of 75 g of enzyme/100 l of solution. The enzyme treatment is carried out for approximately 15 minutes, whereafter the enzyme is inactivated at 70 to 75°C for approximate-

ly 10 minutes.

After the enzyme inactivation, the juice is subjected to an evaporation until it has reached approximately one sixth of the starting amount. The pH value is adjusted to 2.0 by means of HNO_3, and the juice is subjected to a pressure filtration on Celite® 545.

The pressure filtered juice is cooled to approximately 20 to 30°C, and the enzyme treated pectin is precipitated with 1 part of juice to 2 parts of 80% isopropanol (IPA). Then the pectin is processed by passing through a drum screen, whereafter the retentate is carried through a band press.

The molecular weight of the pectin after the enzyme treatment: 33,000 Daltons.

b) Deesterification

The deesterification in a tank reactor uses NaOH in 60% isopropanol at a temperature of 5°C, the NaOH-concentration being 0.3 eq/l.

The pectin material produced above is treated with base for approximately 1 to 2 hours, whereafter a postwashing is carried out with pure 60% isopropanol. Then the material is carried to a band press, and the filtrate is stripped for alcohol in a Lödige-mixer and dried in a drying chamber.

The resulting pectin has a degree of esterification of 4.6%.

Example 2

1 kg of dry pectin with a molecular weight of approximately 100,000 and a degree of esterification of approximately 70% is suspended in 5.7 l of 55% isopropanol comprising 380 ml of 28% NaOH in a tank reactor. The

temperature is adjusted at 60°C and kept there. After 1 hour the suspension is removed and filtered through a cloth, and the extracted solids are washed in 2 times 10 l of 60% isopropanol. Subsequently, the pectin is suspended in 10 l of 60% isopropanol, and the pH value is adjusted to 4.5 to 5.0 by means of 84% phosphoric acid. The pectin is washed again in 10 l of 60% isopropanol and dried.

A pectin with the following characteristics is obtained:

Molecular weight: 35,000 Daltons
DE: 5.2%

Example 3

9 kg of dry pectin with a molecular weight of approximately 110,000 and a degree of esterification of approximately 69% is suspended while stirred in 51 l of 55% IPA admixed 4.2 l of 28% NaOH, and the temperature is adjusted at 45°C.

After 1 hour at this temperature, the pectin is extracted in the same manner as in Example 2.

A pectin with the following characteristics is obtained:

Molecular weight: 28,000 daltons
DE: 1.7%

Example 4

a) Deesterification

22.4 kg of wet pectin obtained in a conventional manner as described in

the above EP 664,300 A1, with a solids content of 25% and with a molecular weight of approximately 90,000 and a degree of esterification of approximately 60% are suspended in 320 l of 55% IPA admixed 2.4 l of 28% NaOH at at temperature of 5°C for 2 hours in a tank reactor.

Degree of esterification of the pectin after base treatment: 3 to 5%
Molecular weight of the pectin after base treatment: 70,000

b) Molecular weight reduction

2.8 kg of dry pectin from step a) are suspended in 16 l of 55% IPA admixed 190 ml of 28% NaOH at a temperature of 60°C in a tank reactor.

The suspension is removed after 2 hours and processed as described in Example 2.

A pectin with the following characteristics is obtained:

Molecular weight: 38,000 Daltons
DE: 2.0%

Example 5

A pectin product is produced which is pumpable and can be dosed, and which can be dissolved in ordinary tap water with a hardness of up to 10°H by way of a simple mixing of the ingredients.

Composition.

Ingredients	Amount %
pectin according to Example 1	70.0%
icing sugar	30.0%

Example 6

A pectin product is produced which is pumpable and can be dosed, and which can be dissolved in ordinary tap water with a hardness of up to 10°H by way of a simple mixing of the ingredients.

Composition:

Ingredients	Amount, %
pectin according to Example 1	52.5%
GENU Pectin type LM-5 CS*	17.5%
icing sugar	30.0%

* Produced by Copenhagen Pectin A/S

Example 7

In analogy to Example 6, a pectin product is produced which is pumpable and can be dosed, and which can be dissolved in water with a hardness of 10°H or more, and which requires no chopping equipment during the production.

Composition:

Ingredients	Amount%
pectin according to Example 2	52.5%
GENU Pectin type LM-5 CS	17.5%
icing sugar	30.0%

Example 8

In analogy to Example 6, a pectin product is produced which is pumpable and can be dosed, and which can be dissolved in water with a hardness of 10°H or more.

Composition:

Ingredients	Amount, %
pectin according to Example 3	35.0%
GENU pectin type LM-5 CS	35.0%
icing sugar	30.0%

Example 9

Like in Example 6, a pectin product is produced which is pumpable and can be dosed, and which can be dissolved in water with a hardness of 10°H or more.

Composition:

Ingredients	Amount, %
pectin according to Example 4	35.0%
GENU pectin type LM-5 CS	35.0%
icing sugar	30.0%

EXAMPLES OF APPLICATION

The following Examples illustrate the use of the above pectin products for the production of bake stable products.

EXAMPLE OF APPLICATION 1

A bake stable jam is produced according to the following recipe.

Order of addition	Ingredients	kg or litres	% of soluble solids (SS)	kg SS
A	Strawberry sauce	20.0	10	2.0
	Saccharose	53.0	100	53.0
	Syrup with a high fructose content	27.6	70	19.3
B	Pectin product acc. Example 5	1.2	100	1.2
	Water	11.0	-	-
C	Citric acid, 50 weight/volume% solution	0.7	50	0.35
	Total ingredients	113.5		75.85
	Evaporation	13.5		
	Yield	100.0		75.9

pH value in product : 3.5 to 3.6

Water activity a_w : 0.70

The production of the bake stable jam is carried out by a method comprising the following steps:

1) the ingredients (A) are mixed,

2) a heating to the boiling point is performed and a boiling is carried out until the content of soluble solids (SS) is 80%,

3) the pectin product according to Example 5 is dissolved in hot water (B) by means of a Silverson L4R mixer with a speed of 6,000 to 8,000 rpm

4) the pectin solution is added to the batch,

5) the content of soluble solids is adjusted to 75% by means of a manual refractometer (45 to 80% of soluble solids),

6) citric acid (C) is added in order to adjust the pH value to 3.5 to 3.6 and

7) a cooling to 70°C is carried out followed by a filling of the product in glasses.

The produced strawberry jam is tested with respect to bake stability by baking for 10 minutes at 200°C. The product turns out to have a satisfactorily bake stability since it does not flow.

The testing of the bake stability involves the following test.

A sample jam of 10 g is placed on filter paper in a metal ring of well-defined dimensions (height 7 mm, diameter 35 mm). Before the placing in an oven, the ring is removed, and after 10 minutes at 200°C, a sample is taken. As a measure of the bake stability, an inner annular diameter is indicated on the filter paper. By an optimum bake stability the jam keeps within this ring.

In addition, the surface must appear smooth. According to an alternative baking test, the jam is placed in the middle of a puff paste slice and baked for 20 minutes at 220°C. The bake stability is visually evaluated.

EXAMPLE OF APPLICATION II

Order of addition	Ingredients	kg or litres	% of soluble solids (SS)	kg SS
A	Strawberry sauce	20.0	10	2.0
	Saccharose	53.0	100	53.0
	Syrup with a high fructose content	27.6	70	19.3
B	Pectin product acc. to Example 6	1.2	100	1.2
	Water	11.0	-	-
C	Citric acid, 50 weight/volume% solution	0.7	50	0.35
	Total ingredients	113.5		75.85
	Evaporation	13.5		
	Yield	100.0		75.9

pH value in product : 3.5 to 3.6

Water activity a_w : 0.70

The jam is produced in the same manner as in example of application I.

The produced strawberry jam is tested with respect to bake stability by baking for 10 minutes at 200°C. The product reveals a good bake stability, and the low water activity ensures a long life and prevents the baked biscuits from turning soft.

EXAMPLE OF APPLICATION III

A bake stable jam is produced according to the following recipe.

Order of addition	Ingredients	kg or litres	% of soluble solids (SS)	kg SS
A	Strawberry sauce	20.0	10	2.0
	Saccharose	53.0	100	53.0
	Syrup with a high fructose content	27.6	70	19.3
B	Pectin product acc. to Example 7	1.2	100	1.2
	Water	11.0	-	-
C	Citric acid, 50 weight/volume% solution	0.7	50	0.35
	Total ingredients	113.5		75.85
	Evaporation	13.5		
	Yield	100.0		76

pH value in product : 3.5 to 3.6

Water activity a_w : 0.70

The jam is produced in the same manner as in example of application I.

The produced strawberry jam is tested with respect to bake stability by baking for 10 minutes at 200°C. The product reveals a good bake stabili-

ty, and the low water activity ensures a long life and prevents the baked biscuits from turning soft.

EXAMPLE OF APPLICATION IV

A bake stable jam is produced according to the following recipe.

Order of addition	Ingredients	kg or litres	% of soluble solids (SS)	kg SS
A	Strawberry sauce	20.0	10	2.0
	Saccharose	48.7	100	48.7
	Syrup with a high fructose content	25.3	70	17.7
B	Pectin product acc. to Example 8	1.2	100	1.2
	Water	11.0	-	-
C	Citric acid, 50 weight/volume% solution	0.7	50	0.35
	Total ingredients	106.9		70.0
	Evaporation	6.9		
	Yield	100.0		70.0

pH value in product : 3.5 to 3.6

water activity a_w : 0.75

The jam is produced in the same manner as in Example of Application I,

whereby, however, in step 2) the solids content is adjusted to approximately 75% and in step 5) the solids content is adjusted to 70%.

The product is tested with respect to bake stability and reveals a good bake stability on filter paper at 200°C for 10 minutes.

EXAMPLE OF APPLICATION V

A bake stable jam is produced according to the following recipe.

Order of addition	Ingredients	kg or litres	% of soluble solids (SS)	kg SS
A	Strawberry sauce	20.0	10	2.0
	Saccharose	45.1	100	45.1
	Syrup with a high fructose content	23.4	70	16.4
B	Pectin product acc. to Example 9	1.1	100	1.1
	Water	20.0	-	-
C	Citric acid, 50 weight/volume% solution	0.7	50	0.35
	Total ingredients	110.3		65.0
	Evaporation	10.4		
	Yield	100.0		65.1

pH value in product : 3.5 to 3.7

Water activity a_w : 0.82

The jam is produced analogously with the method stated in Example of Application I, whereby, however, in step 2) the solids content is adjusted to approximately 70% SS and in step 5) the solids content is adjusted to 65% SS.

The product is bake stable on filter paper at 200°C for 10 minutes.

EXAMPLE OF APPLICATION VI

A bake stable jam is produced according to the following recipe.

Order of addition	Ingredients	kg or litres	% of soluble solids (SS)	kg SS
A	Strawberry sauce	5.0	10	0.5
	Saccharose	53.6	100	53.6
	Syrup with a high fructose content	27.9	70	19.5
	Deionized water	14.1		
B	Pectin product acc. to Example 8	1.0	100	1.0
	Water	11.0	-	-
C	Citric acid, 50 weight/volume% solution	1.3	50	0.65

	Total ingredients	113.9		75.3
	Evaporation	13.9		
	Yield	100.0		70

pH value in product : 3.5 to 3.6

Water activity a_w : 0.75

The jam is produced in the same manner as in example of application I.

The product is tested with respect to bake stability and reveals a good bake stability on filter paper at 200°C for 10 minutes.

EXAMPLE OF APPLICATION VII

A bake stable jam is produced according to the following recipe.

Order of addition	Ingredients	kg or litres	% of soluble solids (SS)	kg SS
A	Strawberry sauce	30.0	10	3.0
	Saccharose	51.7	100	51.7
	Syrup with a high fructose content	26.9	70	18.8
B	Pectin product acc. to Example 8	1.2	100	1.2
	Water	11.0	-	-
C	Citric acid, 50 weight/volume% solution	0.7	50	0.35
	Total ingredients	121.5		75.0
	Evaporation	21.5		
	Yield	100.0		70.0

pH value in product : 3.5 to 3.6

water activity a_w : 0.75

The jam is produced in the same manner as in example of application I.

The product is tested with respect to bake stability and reveals a good bake stability on filter paper at 200°C for 10 minutes.

EXAMPLE OF APPLICATION VIII

A bake stable jam is produced according to the following recipe.

Order of addition	Ingredients	kg or litres	% of soluble solids (SS)	kg SS
A	Raspberry sauce	20.0	10	2.0
	Saccharose	48.7	100	48.7
	Syrup with a high fructose content	25.3	70	17.7
B	Pectin product acc. to Example 8	1.2	100	1.2
	Water	11.0	-	-
C	Citric acid, 50 weight/volume% solution	0.7	50	0.35
	Total ingredients	106.9		70.0
	Evaporation	6.9		
	Yield	100.0		70.0

pH value in product : 3.5 to 3.6

Water activity a_w : 0.75

The jam is produced in the same manner as in Example of Application I, whereby, however, in step 2) the solids content is adjusted to approximately 75% and in step 5) the solids content is adjusted to approximately 70%.

The product is tested with respect to bake stability and reveals a good bake stability on filter paper at 200°C for 10 minutes.

EXAMPLE OF APPLICATION IX

A bake stable hazelnut filling is produced according to the following recipe

Order of addition	Ingredients	kg or litres	% of soluble solids (SS)	kg SS
A	Pectin product acc. to Example 6	0.5	100	0.5
	Hot water	14.0	-	-
B	Syrup with a high fructose content	57.0	70	40
C	Skimmed milk powder	8.0	100	0.8
	Fat-reduced cocoa	6.0	100	6.0
	Vanilla flavour	0.3	100	0.3
	Dry cream flavour	0.3	100	0.3
	Hazelnut paste	14.0	100	14.0
D				
	Total ingredients	100.1		69.1

pH value in product: 6.0

Water activity a_w : 0.8

The production of the bake stable hazelnut filling is carried out by a method comprising the following steps:

1) The pectin product according to Example 6 is dissolved in hot water (ingredients (A)) by means of a Silverson L4R mixer with a speed of 6,000 to 8,000 rpm

2) ingredient (B) is added while stirring,

3) the ingredients (C) are dry mixed and added to the solution, whereafter the mixing continues for 5 minutes,

4) the hazelnut paste is added during the mixing, and

5) a cooling to 70°C is effected, whereafter the product can be filled into for instance bread-like materials, such as croissants.

The produced hazelnut filling has the following properties:

- good bake stability*
- controlled texture
- good release of flavours
- flexible filling temperature
- reduced fat content.

* The bake stability is tested by placing the above product in the middle of a puff paste slice and baking it for 20 minutes at 220°C. The bake stability is visually evaluated.

COMPARISON TEST 1

In the same manner as in Example 2, two pectins are produced with a molecular weight of 10,000 and 18,000, respectively, and a degree of esterification of approximately 10%.

A test of the products with respect to bake stability analogously with the Examples of Application I-VIII revealed that the products were light-fluid and caused syneresis, and that the filling texture was too thin. A baking test on filter paper revealed that the jam flowed 4 to 5 cm beyond the indicated annular diameter.

COMPARISON TEST 2

In the same manner as in Example 2, two pectins are produced with a molecular weight of approximately 18,000 and a degree of esterification of 17% and 19%, respectively.

The low viscosity of the solutions resulted in a poor result for both solutions, the filling being relatively fluid and causing syneresis at the bake stability test. The jam flowed 3 to 4 cm beyond the annular diameter.

COMPARISON TEST 3

A pectin with a molecular weight of approximately 29,000 and a degree of esterification of approximately 24% was produced in the same manner as in Example 2.

The solution was low-viscous and provided an unsatisfactory result by the bake stability test, the filling becoming relatively fluid and causing syneresis.

The jam flowed 3 to 4 cm beyond the annular diameter.

The above description of the invention reveals that it is obvious that it can be varied in many ways. Such variations are not to be considered a deviation from the scope of the invention, and all such modifications which are obvious to persons skilled in the art are also to be considered comprised by the scope of the succeeding claims.

Claims

1. Pectin for use in paste-like materials, c h a r a c t e r i s e d by having a molecular weight in the range of from approximately 20,000 to approximately 50,000 Daltons and a degree of esterification (DE) of less than approximately 20.

2. Pectin as claimed in claim 1, c h a r a c t e r i s e d by having a DE of less than approximately 10, preferably less than approximately 5.

3. Pectin as claimed in claim 1 or 2, c h a r a c t e r i s e d by having a molecular weight in the range of from approximately 25,000 to approximately 40,000 Daltons.

4. A method of producing the pectin as claimed in claims 1 to 3, c h a r a c t e r i s e d by obtaining the pectin in a manner known per se, followed by subjecting the pectin to a molecular weight reduction as well as a reduction of the degree of esterification.

5. A method as claimed in claim 4, c h a r a c t e r i s e d by the molecular weight reduction and the reduction of the degree of esterification being performed in one step.

6. A method as claimed in claim 4, c h a r a c t e r i s e d by the reduction of the degree of esterification and the molecular weight reduction being performed in two steps, preferably by initially reducing the degree of esterification followed by a reduction of the molecular weight.

7. A method as claimed in any of the claims 4 to 6, c h a r a c t e r i s e d by the reduction of the degree of esterification being performed by means of base, such as an alkali metal hydroxide, such as NaOH, KOH or LiOH, or by means of an alkaline earth metal hydroxide, such as $Ca(OH)_2$

or $Mg(OH)_2$, or enzymatically, such as by means of pectin methylesterase, or by means of a combination of an acid treatment with an inorganic acid, such as HNO_3, HCl or H_2SO_4, preferably HNO_3, with an alkaline treatment and/or an enzymatic treatment.

8. A method as claimed in any of the claims 4 to 6, characterised by the molecular weight reduction being performed enzymatically, such as by means of endopolygalacturonase, pectin lyase or pectate lyase, or by means of alkaline treatment, such as by means of an alkali metal hydroxide, especially NaOH, KOH or LiOH, preferably NaOH, or by means of an alkaline earth metal hydroxide, such as $Ca(OH)_2$ or $Mg(OH)_2$.

9. A paste-like material, characterised in that as gelling agent it comprises a pectin as claimed in any of the claims 1 to 3.

10. A paste-like material as claimed in claim 9, characterised in that as gelling agent it comprises a combination of at least one primary pectin as claimed in any of the claims 1 to 3 and at least one secondary pectin with a molecular weight in the range of from approximately 50,000 to approximately 150,000 Daltons and a degree of esterification of less than approximately 20.

11. A paste-like material as claimed in claim 10, in which the secondary pectin has a DE of less than approximately 10, preferably less than approximately 5.

12. A paste-like material as claimed in claims 9 to 11, in which the secondary pectin has a molecular weight in the range of from approximately 60,000 to approximately 110,000 Daltons, preferably from approximately 80,000 to approximately 90,000 Daltons.

13. A paste-like material as claimed in any of the claims 10 to 12, in

which the ratio of primary pectin to secondary pectin is in the range of from approximately 5:1 to approximately 1:3, preferably in the range of from approximately 3:1 to 1:1.

14. The use of a pectin as claimed in any of the claims 1 to 3 for the production of a paste-like material as claimed in any of the claims 9 to 13.

15. The use of a paste-like material as claimed in any of the claims 9 to 13 in a fruit-based product.

16. The use as claimed in claim 15, in which the paste-like material is used in an amount in the range of 0.8 to 1.5% by weight, preferably approximately 1.0 to 1.2% by weight.

17. The use of a paste-like material as claimed in any of the claims 9 to 13 in a milk-based product.

18. The use as claimed in claim 17, in which the paste-like material is used in an amount in the range of 0.8 to 1.5% by weight, preferably approximately 1.0 to 1.2% by weight.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/DK 99/00026

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: C08B 37/06, C08L 5/06, A23L 1/0524
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: C08B, C08L, A23L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI, CAPLUS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 9730093 A1 (HERCULES INCORPORATED), 21 August 1997 (21.08.97), page 5, line 14 - line 26	1-3,9
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X	STN International, File CAPLUS, CAPLUS accession no. 1984:607674, Document no. 101:207674, Aimukhamedova, G.B. et al: "Pectic substances from sea buckthorn"; 11-1 (Plant Biochemistry)	1
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A	WO 8912648 A1 (A/S KOBENHAVNS PEKTINFABRIK), 28 December 1989 (28.12.89)	1-18
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[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" erlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
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INTERNATIONAL SEARCH REPORT

International application No.
PCT/DK 99/00026

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	EP 0580252 A2 (QUEST INTERNATIONAL B.V.), 26 January 1994 (26.01.94), page 2, line 37 - line 41; page 3, line 47 - line 48 --	1-18
A	GB 2145103 A (THE PROCTER & GAMBLE COMPANY), 20 March 1985 (20.03.85), page 5, line 12 - line 16, abstract, claims -- --------	1-13

INTERNATIONAL SEARCH REPORT

Information on patent family members

07/04/99

International application No.
PCT/DK 99/00026

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